Laser-induced chemical modification and nanostructuring: the interface between TWIP steel and PEEK

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Interface preparation for adhesive bonding

Adhesive bonding has become a key technology with many advantages for modern hybrid materials and joining.

Adhesive joining: cheap, no ‚parasitic' excess weight (bolts, rivets…), no drill holes, different material classes can be bonded.

Hybrid laminates: Combining the advantages of two materials, minimizing their respective short-comings.
E.g. Ti alloys + PEEK: A thermoplastic matrix, remeltable, reshapable, chemically inert:

- Problem: unreactive = also no formation of chemical bonds with most surfaces!

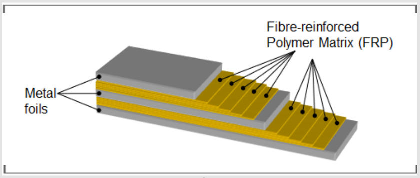

Fig.1: Schematic lay-up of a hybrid material based on adhesive bonding: a laminate with fiber-reinforced polymer matrix, e.g. Titanium/CF-PEEK.

Laser Parameter Study

1. Materials:

- Thermoplastic PEEK (Polyether ether ketone 100µm film): high temperature stability, very high tensile toughness.

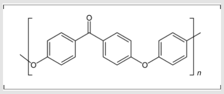

Fig. 2: Molecular functionalities of a PEEK monomer.

- Metal: Fe-Mn-Al-Si alloy, TWIP steel, 72.5×10×1.6 mm³
- 6 samples per laser parameter set

2. Laser pretreatment in air:

Cleanlaser CL20:

- Line distance 0.03mm, velocity 4000 mm/s
- Power P = 6.6, 13.2, and 20 W!
- Pulse ν = 40, 70, and 100 KHz
- Repetitions R = 1, 5, and 10

  ⇒ Total fluences: 2- 150 J/cm²

3. Sample preparation and aging:

- Single-lap shear, 5×10 mm² joining area (reduced from DIN EN 1465)
- Curing: ~400°C,15 min
- Aging: 3 d in H_2O at 80°C

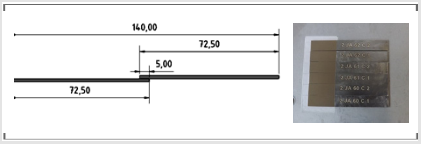

Fig.3: Lap-shear specimen, and photo of laser treated metal stripes.

4. Mechanical testing: Single-lap shear:

- Load: superposition of mode I and mode II
- Approximation:

  Shear strength ≈ joint strength

Results

5. Shear strengths and Degradation:

A „Design of Experiment" analysis was conducted for all 3×3×3 laser parameter sets:

⇒ P and ν: are main factors determining initial and residual strength (Fig. 4)

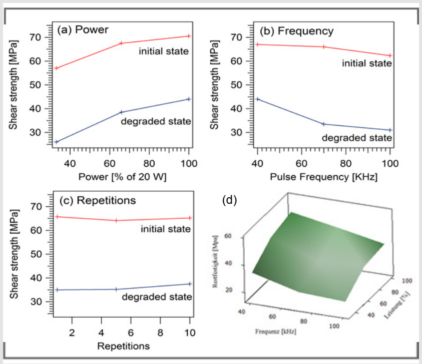

Fig.4: (a)-(c) Main effects plots of power, frequency and repetitions on initial and residual shear strengths, (d) corresponding surface plot.

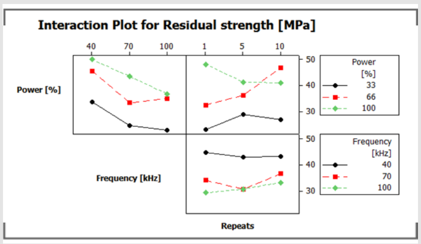

Fig. 5: Interaction between the laser parameters.

⇒ R: on average insignificant, but for low P or high ν a larger number of repetitions (e.g. 10x for 33%, 100 KHz) needed!

⇒ Strong correlation between power and repeats at low to medium powers (Fig. 5).

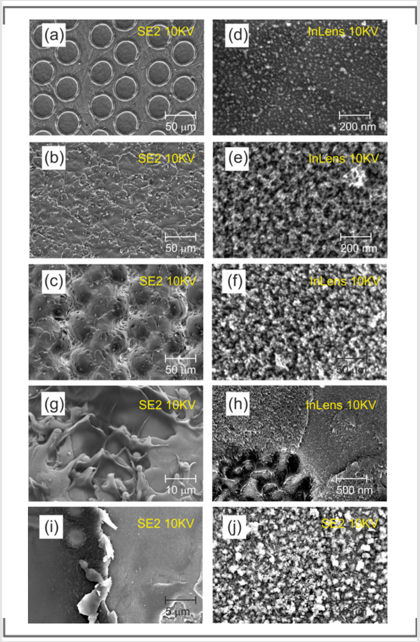

Fig. 6: Pretreatment with 33%, 66% or 100% power, 40KHz, 1x repetition: (a)-(f). (g)-(h) macro and micro structure of a fracture surface covered with PEEK. (i) PEEK rim of at adhesion defect. (j) lasered surface after corrosion.

6. Morpholgy of treated surfaces:

- High fluences: strong macro- and fine nano-structuring (Fig. 6)!
- EDX: mixture of Fe, Mn, Al and Si oxides?
- Repetitions at high fluence:

  ⇒ small decrease of nano-structures.

- Massive corrosion on aged specimen.

6. Fracture surfaces and adhesion defects

20% of all samples, particulary those treated at high fluences, showed PEEK degradation and bonding failure!

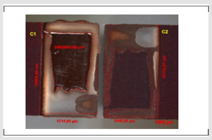

Fig. 7: Adhesive defect observed after fracture.

- Areas were mostly in center, connected by channels to the outside: gas evolution?
- Discoloration: Interference effects or chemical species?

⇒ Reactions? E.g. redox, Mn^{d+} d>>2

Conclusions

⇒ Max. St_{ini} = 75.3 MPa (100%, 40KHz, 5x)

⇒ Max. St_{res} = 56.5 MPa (100%, 40KHz, 1x)

Low number of repeats suffice, but high power and low pulse frequency required to create sufficient nano-structuring that ensures long-term stable, strong bonding.

Corrosion and adhesive defects are major problems encountered with TWIP steel!

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